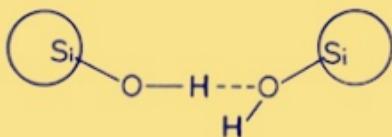


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FUMED SILICA



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FUMED SILICA

The element silicon is widespread throughout the world being the second most abundant element in the earth's crust. It is the fundamental part of most minerals and is present in one form or another in all rocks, clays and soils.

Silicon (Si) is never found free in nature but is almost always combined with oxygen atoms. (Fig. 1a).

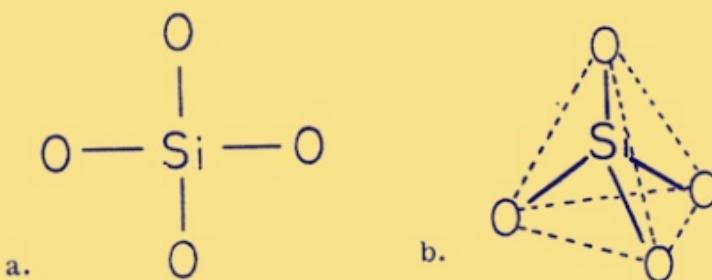


Fig. 1

The four oxygen atoms which attach themselves to a silicon atom are arranged in a tetrahedron, a four-sided geometric shape, around the silicon atom (Fig. 1b).

One of the characteristics of silicon is its tendency to form long chains. In most rocks and minerals, the silicon tetrahedra are linked together in various ways in a lattice network.

One of the most widely distributed

forms of silicon is silicon dioxide, or silica, SiO_2 . Silica is found in pure form in nature as quartz. Sand, flint, sandstone and opal are other examples of silica minerals, but contain traces of impurities.

Silica does not exist as separate molecules of SiO_2 , but consists of an interlocking structure of silicon and oxygen atoms bonded together tetrahedrally. Each oxygen atom of a tetrahedron is linked to a silicon atom in an adjacent tetrahedron and is thus a member of a second silicon tetrahedron (Fig. 2).

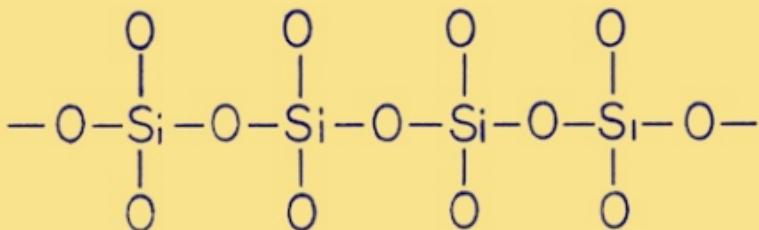


Fig. 2

The formula, SiO_2 , therefore, merely represents the ratio of the two elements present in silica—two oxygen atoms to each silicon atom.

Countless numbers of such tetrahedra are linked together tightly in each tiny grain of sand. The bond between the oxy-

gen and silicon atoms is very strong and difficult to sever making such minerals as quartz and sand very hard and inert, and capable of withstanding very high temperatures.

You are all familiar with the hard crystalline structure of sand and its sharp contours that make it useful for such purposes as sand paper and in scouring powders.

The powder in your unit is also a silica, but as you can see quite different in appearance from sand. This silica is fumed silica. In chemical composition, it is essentially pure silica, SiO_2 . It is chemically inert and can be heated to high temperatures without melting. But because it has been prepared in a special way, it has acquired some unusual properties not characteristic of crystalline silica, making it useful in a variety of different applications.

With the materials in this unit, you will demonstrate some of these interesting properties and learn why fumed silica behaves as it does.

First, examine your specimens.

FUMED SILICA—Fine white powder in polyethylene bag.

CONE-SHAPED CUP—To test free-flow of powders.

KRAFT PAPER—Two pieces.

PROTRACTOR—To be cut out for use.

WHAT IS FUMED SILICA?

Fumed silica is an inert and very fine powder consisting of submicroscopic particles made up of many short chains of silica molecules joined together by fusion during the manufacturing process and by hydrogen bonding (Fig. 3).

The fineness of the particles, much finer than the tiniest grains of sand, and their

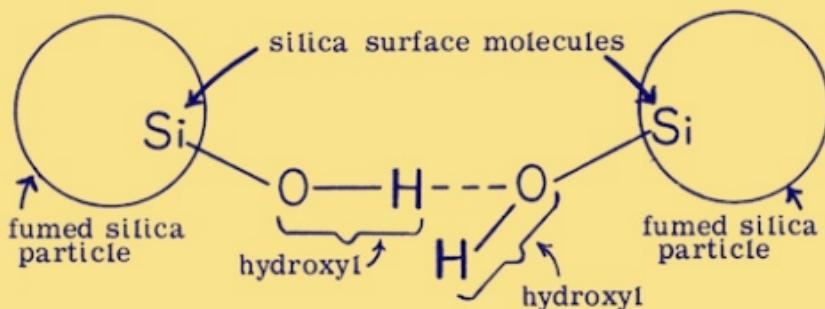


Fig. 3

complexity gives fumed silica an enormous external surface and provides a great quantity of surface molecules for reaction. Surface molecules are those that are not completely surrounded by similar

molecules (Fig. 3). An increase in surface molecules is always accompanied by changes in the physical properties of a substance. This is what occurs in the molecules of fumed silica.

Most of the surface silica molecules on the particles of fumed silica have a hydrogen atom attached to one of its oxygen atoms forming a hydroxyl or -OH group (Fig. 3). This peculiarity of the silicon tetrahedron gives fumed silica properties not evident in usual silica crystals, such as sand and quartz.

A hydrogen atom on one fumed silica particle is attracted to an oxygen atom on another particle. If the particles come close enough to each other, the hydrogen and oxygen atoms form a bond and the particles form short chains. This process is known as hydrogen-bonding. In Fig. 3, the dotted line indicates where the hydrogen-bonding takes place.

PROPERTIES OF FUMED SILICA

Experiment 1. Hold the bag of white powder in your unit in the palm of your hand and notice how light it is. The bag contains about one-half ounce of fumed silica. It is much lighter than sand

as you can see. A tablespoon of sand would weigh about the same as the bag of fumed silica.

Experiment 2. Open the bag of fumed silica very carefully by cutting it across the top with a pair of scissors. Handle it gently. The silica is so fine and light it will scatter easily.

In all your experiments handle the powder with care to prevent it from flying around.

Take a pinch of the fumed silica and rub it between your fingers. Note the dry feeling it causes. The substance has a wicking effect, drawing up the moisture and oil from your fingertips. This property makes it very useful in keeping various materials dry and free-flowing in humid weather. Fumed silica attracts moisture and is hydrophilic.

Experiment 3. Place a tablespoon of water in a small covered jar or vial. Add to this a pinch of the fumed silica. Cover the jar and shake the mixture vigorously.

What happens to the silica?

Does it become dispersed throughout the liquid? Does it appear to go into

solution? Silica is not soluble in water and its particles do not go into solution but are dispersed, and because of their fine size form a colloidal suspension. Substances forming colloidal suspensions do not dissolve in the liquid in which they are dispersed.

Experiment 4. Gradually add more of the silica to the mixture, $\frac{1}{4}$ teaspoon at a time, shaking well after each addition, until the mixture thickens into a stiff gel. Make a record of the amount of fumed silica added.

When a very small amount of the silica is dispersed in water, its short-chain particles spread throughout the liquid in a suspension and are too far apart to attract each other for hydrogen bonding. In addition, hydrogen bonding between the water and silica takes place further preventing network formation.

However, as more of the powder is added, there is a greater chance of contact between the short chains and hydrogen bonding between them can take place increasingly. The mixture gradually becomes thickened and more viscous. When the concentration of the silica is increased

sufficiently, the short chains form a tight network structure, preventing water from flowing freely within the system, and a gel state results. Figure 4 shows graphically how the short chains become linked together to form a network.

The gel formation of fumed silica is influenced by the nature of the water used. Therefore, depending upon the

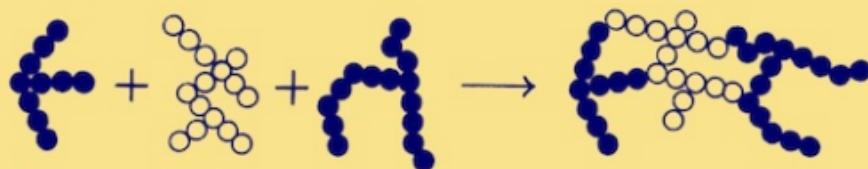


Fig. 4

water in your locality, you may need to use slightly more or less powder than the amounts given.

In the gel state of a colloidal system the solid is arranged in a fine network that extends throughout the liquid and both the solid and liquid phases are continuous.

Experiment 5. Shake the gelled mixture. Does it become liquid again? Now allow it to rest. Does it gel again?

When the mixture is agitated, the net-

work formed by the short chains is broken down allowing the water to flow more freely again. This is the sol state of a colloidal system in which the solid dispersed through a liquid is discontinuous or broken and the liquid forms a continuous phase.

When the agitation is stopped, the attraction between the hydrogen and oxygen atoms causes them to reform the network and assume the gel state once more.

This property that enables certain colloidal gels to liquefy when agitated and revert to a gel when at rest, is known as thixotropy.

The ability of fumed silica to induce thixotropy makes it useful in such applications as eliminating slump, sag or run in resins, sealants, adhesives and paints.

The gel system can be destroyed by diluting the mixture. Add a teaspoon or so of water to the gel and note what happens.

Experiment 6. If you have some turpentine or mineral oil, take a tablespoon of the liquid and gradually add fumed silica to it as you did with water. What happens? Does it gel?

Do you need more or less of the silica to produce gelling of turpentine or mineral oil than to produce the same effect in water?

Is the gel clear? How does it compare with the gel formed with water?

Shake the mixture in a small jar. Is it thixotropic also?

Shake it again and then turn the container upside down. Does the gel drip? The mixture assumes its gel state as soon as the agitation stops and remains at the bottom of the container. Thixotropic agents such as fumed silica are important additives in the manufacture of dripless paints and greases.

Experiment 7. Place a small button or other object in the jar of gelled turpentine or mineral oil. Shake the jar and note that the button moves about freely in the mixture.

Now let the mixture rest. Does the object sink to the bottom? It remains suspended. Turn it gently upside down. The button does not change its position. It is held tightly in the gel. As soon as you agitate the mixture, it will move about again.

In industry, to obtain a higher viscosity and thixotropy an additive may be used. These additives act as bridging agents that form additional links between the silica chains.

The gelling ability of a mixture of a liquid and fumed silica depends primarily on the polarity of the particular liquid being used.

What is polarity?

When chemical reactions take place, the atoms may combine by either ionic bonding or covalent bonding. In ionic bonding, electrons are transferred from one atom to another, while in covalent bonding two atoms share their electrons with each other and no transference of electrons occurs between the two.

If the two atoms that share their electrons are alike, each atom will attract the electrons equally. For example, hydrogen gas, H_2 , is formed by covalent bonding, H-H. The bond is then said to be symmetrical and the electrons are distributed equally about both atoms. Electrically symmetrical bonds are nonpolar.

However, if the compounds are formed by covalent bonding of unlike atoms, they

most likely will differ in their attraction for the pair of bonding electrons, and the electrons will not be equally shared. They will be held more closely by the atom having the greater attractive force and this atom will acquire a more negative charge and the other a more positive charge. This relative attraction of an atom for bonding electrons is referred to as the electronegativity of the atom. The bond between the two therefore would be unsymmetrical and the bond is called a polar covalent bond and the compound is said to be polar. The degree of polarity differs from substance to substance.

In water, the bonds between the oxygen and hydrogen are not equally shared, the oxygen being more strongly attracted and therefore somewhat negative and the hydrogen as a result is somewhat positive. Water has two covalent hydrogen-oxygen bonds which are unsymmetrically arranged in the molecule. Water, therefore, is a polar compound.

The polarity of a liquid affects its ability to form hydrogen bonds.

If the liquid in which the fumed silica is dispersed is highly polar, it will form bonds with the -OH group of the silica

interfering with the linkage of the silica chains with each other.

Since the greatest thickening efficiency with the silica is obtained when the -OH groups on the surface can bond with each other and form a network structure, in a mixture of fumed silica and a liquid, the more polar a liquid is, the greater the amount of fumed silica required to form a gel.

Water is a polar substance while turpentine is nonpolar. Therefore, in your experiments a gel was produced with less powder when turpentine was used than when water was the medium.

Experiment 8. Add silica to a tablespoon of vinegar. Compare the amount of silica that must be added to the vinegar to cause it to gel with the quantities that gelled water and turpentine.

Liquids such as turpentine and mineral oil which have little or no polarity and do not bond with hydrogen, generally require 1% to 3% by weight of the fumed silica of the type in your unit to form a gel.

Acetic acid (vinegar) is highly polar and requires much more of the silica to produce a gel.

Fumed silica itself is polar in nature and in nonpolar, non-hydrogen-bonding liquids, the hydroxyl groups are actually repelled by the components of the fluid causing the fumed silica chains to migrate toward each other thus hastening the formation of a network.

Experiment 9. Place a teaspoon of vinegar in a small deep dish like a custard cup and to this add 2 tablespoons of the fumed silica. Stir it well to disperse the particles evenly. If the mixture is damp add a little more of the powder. Stir until the particles are very fine and there are no lumps. Continue to add the silica until the mixture looks dry. You have transformed liquid vinegar into a solid.

Smell the powder. It will smell just like vinegar since its chemical composition has not been changed.

Very high concentrations of fumed silica convert liquids into a free-flowing powder.

You can convert water into a solid in the same way

The process which takes place is responsible for the dried feeling that you sensed when you rubbed the powder be-

tween your fingers.

The fumed silica gels the liquid first, but as more and more of the powder is added and the mixture is stirred, the liquid separates into smaller and smaller droplets and the short chains of silica particles form tight networks about each little droplet completely surrounding them. The free-flowing powdered vinegar, therefore is actually composed of droplets of vinegar enclosed in fumed silica.

Experiment 10. Add a quarter of a teaspoon of the fumed silica to a tablespoon of 20-30-weight non-detergent motor oil and stir it in well. Does it thicken into a grease? Add a little more if necessary until it becomes paste-like. You have converted an oil into a grease.

Fumed silica is used as an additive to greases to give them body.

As with water, the fumed silica network prevents the oil from flowing freely causing it to become thick and viscous.

If you do not have motor oil on hand, try machine oil, such as Three-in-One. You may need to add more silica to achieve the same effect.

When added to foods, fumed silica is used only in minute quantities. For ex-

perimental purposes, the amounts used here are greater than ordinarily used in foods. Therefore DO NOT eat any of your experimental material nor the fumed silica itself.

Experiment 11. Soften a $\frac{3}{8}$ -inch slice of butter or margarine by allowing it to stand at room temperature. Then stir two teaspoons of the silica powder into it mixing together well so that the particles will be dispersed evenly throughout the fat.

Shape the butter back into its original rectangular shape as closely as possible and label it. Allow it to harden in the refrigerator

Construct two shallow cups of the same size out of aluminum foil. After the treated butter has hardened place it and another slice of butter in the cups. Place the two cups into a pan and warm them at a low heat.

Which piece of butter begins to melt first? Continue warming for a minute or two. Which melts more extensively?

The addition of the silica causes the fat to be more resistant to heat. Why?

The network formed by the fumed

silica remains intact even when heated. Therefore, when the butter is heated and reaches a temperature range at which it would normally melt and flow, the fumed silica network interferes with it. As a result the butter retains its stability.

Throw away the treated butter after experimenting.

This property of fumed silica is made use of in bearing lubricants. They can be made to withstand higher speeds, pressures and temperatures without liquefying or oozing out.

Experiment 12. Place two tablespoons of regular flour in two separate small containers. To one add $\frac{1}{8}$ teaspoon of the fumed silica and mix it thoroughly.

Cut the tip off the paper cup in your unit to make a $\frac{1}{4}$ -inch-diameter hole. This will serve as a funnel to observe the ease of flow of the flour.

In industry, the free-flow characteristic of materials is determined by what is known as the "angle of repose." The angle of repose is determined by pouring a material through a cone placed an inch above a flat surface and observing the angle of incline of the mound of material

with the horizontal (Fig. 5). The smaller the angle of repose is, the more free-flowing the material.

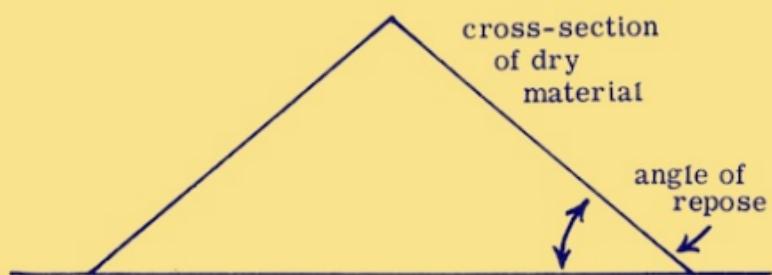


Fig. 5

Pour the untreated flour into the funnel held about an inch above a plate. Does the flour flow readily through the hole? If necessary tap the funnel slightly to assist the flow. Note the shape of the mound formed as the flour flows into the plate.

Now pour the treated flour into the funnel holding it about an inch above a second plate. Note how much more freely the flour flows when it has been mixed with the silica. Compare the angles formed by the two mounds. Which forms the more acute angle? Cut out your protractor and use it to measure the angles of repose.

The submicroscopic size of the fumed silica particles permits them to nest be-

tween larger size particles such as those of the flour, thus separating the flour particles from each other and allowing them to flow without interfering with each other.

Does adding a little more silica powder increase the free-flow of the flour? Check the angle of repose. Is it greater or less when more silica is added?

By testing the effects of the addition of varying amounts of fumed silica to a product, the optimum concentration can be determined for any particular substance by measuring the angle of repose.

Usually one gram or less of fumed silica is used for every 100 grams of caky material.

Throw out the treated flour after you have finished your experiments.

When added to water soluble powders or crystals such as inorganic salts and hygroscopic (water attracting) food crystals, fumed silica forms a protective network or coating on each crystal to prevent particles from sticking together or caking.

Fumed silica is used to maintain free flow in materials such as sulfur, wet lead oxide, table salt, sodium bicarbonate and instant food mixes.

Experiment 13. Can you devise an experiment to show how the quantity of fumed silica added affects the free-flow ability of a substance such as sodium carbonate or brown sugar?

Experiment 14. Place one tablespoon of salad oil and one tablespoon of water in a covered jar and shake the mixture vigorously, or beat the mixture with an egg beater, to reduce the oil to droplets as fine as possible. Then place the mixture aside to rest. Does the oil separate and float to the surface?

Now add $\frac{1}{2}$ teaspoon of fumed silica to the mixture and beat the mixture again.

What happens on standing this time? Do the oil and water remain mixed together?

The silica added forms a network and prevents the tiny droplets of oil from joining together into larger droplets, keeping them separated and suspended in the water. As a result the oil remains emulsified. An emulsion is a colloidal system in which one liquid is dispersed through another liquid. Milk is an example of a colloidal emulsion.

In your experiment the mixture may not remain emulsified if the oil particles are not fine enough.

Experiment 15. Change the proportions, using two tablespoons of water and one tablespoon of oil. Add the same amount of fumed silica and repeat the procedure.

What are your results? Does the oil remain emulsified? If not, does the oil and water mixture rise to the surface?

Since water is a polar liquid and oil ranges from low to medium polarity, in order to produce a complete stable emulsion with such a mixture, would you need more or less silica than used in Experiment 14?

If the amount of fumed silica added is not sufficient to completely emulsify the mixture, the emulsified portion and excess oil will float at the top. Does this happen in Experiment 15?

Experiment 16. Mix two tablespoons of oil, one tablespoon of water and two teaspoons of fumed silica and repeat the experiment.

When the quantity of oil is greater than that of water and the mixture does

not become completely emulsified because too much fumed silica has been added, then the water and oil mixture will sink to the bottom of the container.

Fumed silica is used as an emulsifier and stabilizer in many products including cattle food supplement and tile cleaner.

Experiment 17. Mix together 4 tablespoons of water and 2 teaspoons of fumed silica in a shallow bowl. Place one of your pieces of brown Kraft paper in the mixture and allow it to soak in it for an hour or two. Then remove it and hang it up and allow it to dry thoroughly. It must be absolutely dry for the results of this experiment to be effectively demonstrated.

Take the other piece of Kraft paper and make a mark on it to differentiate it from the treated piece.

Fold both pieces crosswise at the center.

First, take the untreated paper and rub the free ends of the folded piece together. Note that they slip over each other fairly readily.

Repeat with the folded treated paper. Do you notice a definite resistance to slippage?

Things of science
FUMED SILICA

Fumed silica coated on the surfaces of multiwall paper bags and container boards acts as an anti-slip agent. The anti-slip property is made use of in waxes, polishes, plastics, inks and packaging materials. It is also useful in providing a tighter weave in textiles. How is this possible?

The applications of fumed silica are wide and varied. Can you infer from your experiments with the product why fumed silica is useful in helping to increase the tear resistance of rubber, to disperse pigments, to make better printing inks, for adding gloss to polishes, in improving the stability and other qualities of plastics, for preventing caking in cosmetics, as a suspending agent and tablet binder in pharmaceutical products and in providing better flow control in adhesives and sealants?

This unit was prepared in cooperation with Cabot Corporation, Boston, Mass., who supplied the fumed silica for your experimentation.

* * *

Cut out label on other side and put on end of box

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12 monthly experimental kits — \$9.50
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